PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Zaki *et al.*

Group Art Unit: 1751

Serial No.: 10/791,427

Examiner: Gregory R. Delcotto

Filed: March 2, 2004

Docket No.: 1456/3

Confirmation No.: 3775

For: SOLVENT COMPOSITIONS FOR REMOVING PETROLEUM RESIDUE FROM A SUBSTRATE AND METHODS OF USE THEREOF

* *

DECLARATION OF ROBERT E. TROXLER, PH.D.
PURSUANT TO 37 C.F.R. §1.132

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

1. My name is Robert E. Troxler, Ph.D., and I am the Director of Advanced Technologies at Troxler Electronic Laboratories, Inc., of Research Triangle Park, North Carolina, and a co-inventor of the subject U.S. Patent Application Serial No. 10/791,427.

2. A true and accurate copy of my *curriculum vitae*, which evidences my expertise and credentials, is attached herewith and labeled **Exhibit A**.

3. I have had an opportunity to review pending claims 1-43 and 105-110 in the subject above captioned U.S. Patent Application Serial No. 10/791,427.

4. I have also reviewed the following documents: the Non-Final Official Action dated June 23, 2008 on the above captioned U.S. Patent Application Serial No. 10/791,427 by the U.S. Patent and Trademark Office (USPTO); U.S. Patent No.

6,281,189 to Heimann *et al.* (hereinafter the "Heimann Patent"); U.S. Patent Application Publication No. 2003/0213747 of Carbonell *et al.* (hereinafter the "Carbonell Application"); and U.S. Patent No. 5,143,639 to Krawack (hereinafter the "Krawack Patent").

5. The subject matter of the pending claims relates to compositions that can be used to very effectively remove petroleum residue from various substrates such as tools and truck beds.

6. Despite the assertion presented in the Heimann Patent regarding the utility of the compositions disclosed therein to remove asphalt, methyl soyate alone is known to be a very poor asphalt solvent.

7. Biodiesel, as a proxy for methyl soyate, was tested as an asphalt solvent by me using the following procedure, which is a modification of a procedure described in Kulkarni *et al.* (2003) 31 *Journal of Testing and Evaluation* 429-437 (a true and accurate copy of which is attached as **Exhibit B**). 1.5 g of asphalt (Citgo PG-64-22, TK-109 Lot 5) was placed into an aluminum dish, and 0.5 g of solvent was added to the dish. After 5 minutes, the dish was inverted for 5 minutes to drain the solvent and dissolved asphalt. The dish was then rinsed for 5 minutes and dried overnight. The next morning, the dried dish was weighed and the mass of asphalt dissolved was recorded (*i.e.*, the difference between the weight of the aluminum dish plus asphalt before the solvent was added to the total weight after drying overnight). The amount of asphalt dissolved was expressed as a percent of the total asphalt employed (*i.e.*, 1.5 g). Also tested were 100% isopropyl benzoic acid ester (IPB) and diesel fuel, for comparison.

8. The following data were generated. 100% Diesel fuel dissolved 16% of the asphalt, 100% biodiesel dissolved 1.07% of the asphalt, 100% IPB dissolved 46.9%, and surfactant alone (WITCONOL™ 550) dissolved 0.00%.

9. 54% IPB/ 40% biodiesel/ 6% WITCONOL™ 550 dissolved 42.7% of the asphalt. Given the percentages of the various components in the mixture, 54% IPB/ 40% biodiesel/ 6% WITCONOL™ 550 might have been expected to dissolve about 26% of the asphalt under these conditions $[(0.54 \times 46.9) + (0.4 \times 1.06) + 0.06 \times (0.00) = 25.3 + 0.424 + 0.00 = 25.7\%]$.

10. Thus, upon information and belief, mixing an aliphatic ester (*e.g.*, biodiesel or methyl soyate) and an aromatic ester (*e.g.*, IPB) provides a composition that has greater than additive solvent properties with respect to asphalt. It is believed that this result is evidence of a superior property of the claimed compositions that could not have been predicted based on the disclosures of any of the Heimann Patent, the Carbonell Application, and the Krawack Patent, either alone or in combination.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Robert E. Troxler, Ph.D.

Robert E. Troxler, Ph.D.

Dec. 23, 2008

Date

Attachments: **Exhibits A** and **B**

EXHIBIT B

Journal of Testing and Evaluation, Sept. 2003, Vol. 31, No. 5
Paper ID JTE11722_315
Available online at: www.astm.org

Moreshwar Kulkarni,[1] *Qingxia Xu*,[1] *Akhtarhusein Tayebali*[2]

Development of a Procedure to Determine the Effectiveness of Asphalt-Removing Solvents*

ABSTRACT: Due to environmental protection requirements, most state and federal agencies are now required to use biodegradable solvents instead of diesel fuel or other hydrocarbon solvents. A variety of environmentally friendly and safe asphalt-removing solvents are available in the market. However, there is no quantitative standardized procedure to compare the efficacy of these solvents. This paper outlines the development of a procedure used to evaluate the cleaning efficacy of various solvents. The goal was to develop a standardized procedure that would yield quantitative and repeatable results. After evaluating various alternatives like metal and glass plates, ceramic tiles, and aluminum foils, the aluminum dish was found most suitable for the study. Test results obtained for solvent comparison were found to be consistent and repeatable, with the coefficient of variation for asphalt removed less than 10% for most solvents. Further, this study provides an outline for cost-effective analysis of solvents used in relation to diesel fuel and the procedure is able to rank solvents quantitatively.

KEYWORDS: solvents, asphalt, asphalt-removing solvents, emulsion, diesel fuel, CRS-2

Nomenclature

CRS-2 - Cationic rapid-setting asphalt water emulsion
EPA - Environmental Protection Agency
MSDS - Material Safety Data Sheet
NCDOT - North Carolina Department of Transportation

Background

The use of asphalt-removing solvents has been prevalent in the pavement construction industry. Traditionally, asphalt removers were either petroleum distillates or chlorinated solvents or a combination of the two. Safe usage and disposal of these solvents is a big issue due to nonbiodegradability, and the presence of volatile organic compounds and ozone-depleting chemicals. Heightened environmental concerns, stringent EPA rules, and increased disposal costs have encouraged the search of viable alternatives. In 1991, North Carolina Department of Transportation (NCDOT) field units were directed to use biodegradable solvents, instead of diesel fuel, to remove asphalt from tools and equipment. Similar requirements [1]–nonhazardous, vide U.S. regulation Title 40 CFR Part 261 [3]–exist for the Missouri Department of Transportation, and perhaps other state transportation departments as well.

Over the past decade, various solvent products have been available in the market. Most of the commercially available products are either orange, pine, or soy-based solvents and are marketed as "environmentally friendly, safe, and effective." In the past, approving these products for use by NCDOT involved only review of the MSDS and a field trial of a product sample [4]. However, the procedure did not specifically address the effectiveness of dissolving or removing asphalt from paving tools, nozzles, or other equipment nor could it identify solvents based on cost efficiency. Although different empirical solubility scales such as kauri-butanol value (ASTM D 1133 - Standard Test Method for Kauri-Butanol Value of Hydrocarbon Solvents) and aniline point (ASTM D 611 - Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents) exist for hydrocarbons, no solubility testing procedure exists for evaluating specifically a solvent's effectiveness in dissolving asphalt.

Objective and Scope of the Study

The objective of this research study was to develop a standardized, reproducible, and quantifiable test method to evaluate a particular solvent product's effectiveness in dissolving and removing asphalt. The test method, if acceptable, was to be used by NCDOT to evaluate asphalt solvents for the purchase of the most effective and cost efficient products available.

Materials Used and Exploratory Study

Materials Used

Ten commercially available asphalt-removing solvents were evaluated in this study.[3] The solvents were designated S1–S10. Diesel fuel was used as a control solvent. The asphalt used was a residue from CRS-2 emulsion. In addition, the following were used: glass plates, standard bathroom ceramic tiles, aluminum plates, aluminum foils, aluminum dishes, spray bottles, droppers, and a weighing scale with least count of 0.001 g.

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[3] See Appendix

* The contents of this paper reflect the views and opinions of the author(s) and are not necessarily the views of North Carolina State University. The author(s) are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of either the North Carolina Department of Transportation or the Federal Highway Administration at the time of publication. This paper does not constitute a standard, specification, or regulation. This paper does not address any safety issues associated with the use of any of the materials listed in it.

EXHIBIT B

Exploratory Study

During the initial stages of this study, the focus was to evaluate the cleaning efficacy of solvents based on residual asphalt on glass and metal plates, each of size 10 cm by 10 cm. The glass plates needed tempering in order to withstand higher temperatures. Not only is tempered glass expensive, but broken glass poses a safety issue. It was, therefore, decided to use ceramic tiles. Although the tile area initially was covered uniformly with CRS-2 emulsion, the distribution of residual asphalt after baking was nonuniform. The area coated with residual asphalt tended to shrink unevenly. However, asphalt bonded well with the aluminum plates and these were used for the preliminary study.

Initially, the square metal plates were cleaned, dried, and weighed. Then a coat of emulsified asphalt (CRS-2) was applied with a brush onto one side of the plate. The weight of the plates after applying the coat was recorded and the plates were heated in a convection oven for 24 h at 60°C. The heating caused the emulsion to break and, after cooling, the amount of residual asphalt on each plate was determined. Each plate was then sprayed with a different solvent, taking care that the same amount of solvent was sprayed on all plates. After draining the plates for five min, the plates were wiped dry with a paper towel and weighed. The loss of residual asphalt was calculated.

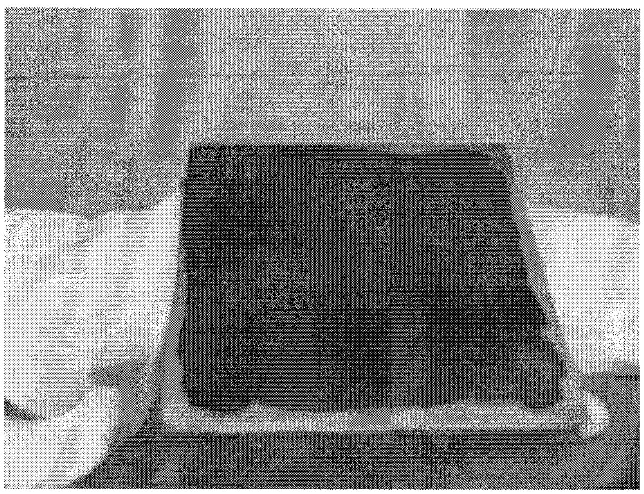

FIG. 1—*Exploratory study, metal plates covered with asphalt.*

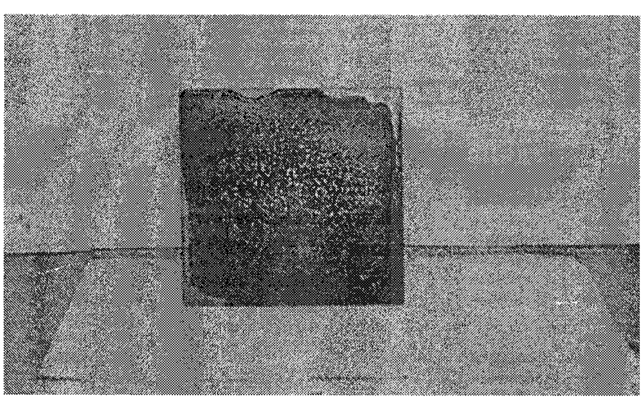

FIG. 2*a*—*Metal plates cleaned with diesel fuel.*

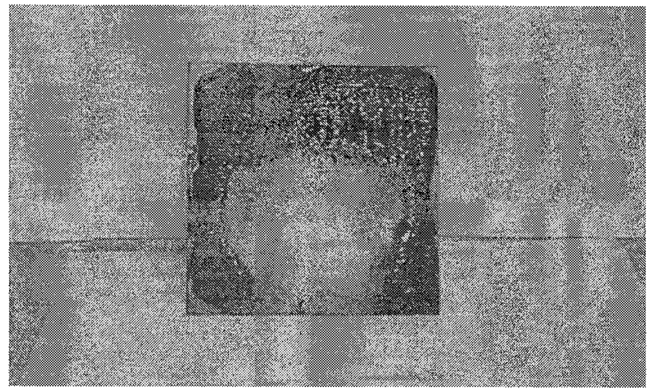

FIG. 2*b*—*Metal plates cleaned with solvent S1.*

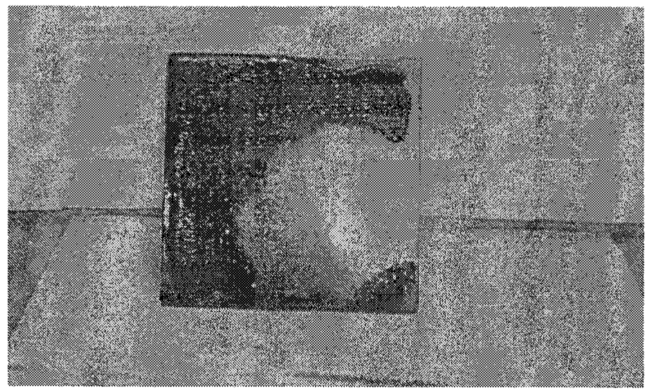

FIG. 2*c*—*Metal plates cleaned with solvent S2.*

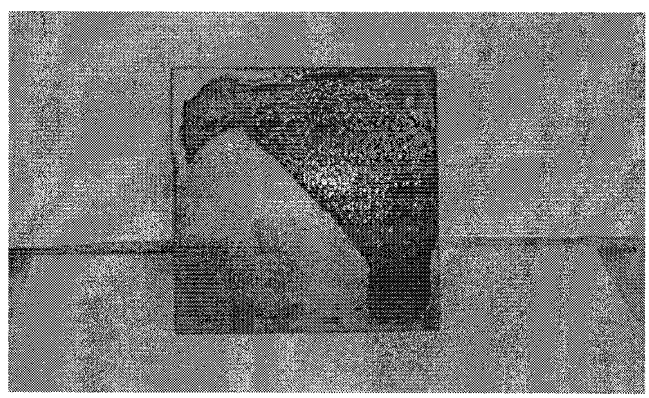

FIG. 2*d*—*Metal plates cleaned with solvent S4.*

Figures 1 and 2*a–d* show visual representations of the procedure. Results indicated that there were several problems associated with the testing procedure and it turned out that the methodology was more qualitative than quantitative. Specifically, the problems encountered were the following:

- when applying an asphalt coat on the plate surface, there was little control over asphalt coat thickness and area of application. Use of a film applicator could give accurate results but this approach was not verified in the current study

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- when heating the plate in the oven, the asphalt tended to flow over the edge of the plate
- while spraying the solvent, it was difficult to control the amount of solvent as well as the uniformity of the application
- the process of draining and cleaning with a paper towel was variable and highly operator specific
- accurate measurement of the asphalt removed was very difficult due to the disparity in the weight of the metal plates compared with the few grams of asphalt used

In spite of all the variability in accurately measuring the amount of asphalt removed, it was possible to determine qualitatively the relative effectiveness of solvents based on visual inspection. Figs. 2a–d show typical results obtained with various solvents.

Use of Aluminum Foil Versus Weighing Dish

In order to reduce the variability, one modification was to use lighter plates so that the measurements could be obtained with better accuracy. The heavy metal plate, therefore, was replaced by lighter aluminum foil and by a weighing dish [2]. In addition, the sidewalls would prevent the residual asphalt from running off, reducing the potential for error. Although there was variability in the weight of aluminum foils, the aluminum weighing dishes had a constant weight of approximately 1 g. The advantage of using the weighing dish was that the bottom surface area of the dish was constant. A fixed amount of asphalt spread over a constant area would give a uniform rate of application, increasing repeatability. Figures 3*a* and *b* show the aluminum foil compared to the aluminum dish used for the rest of this study.

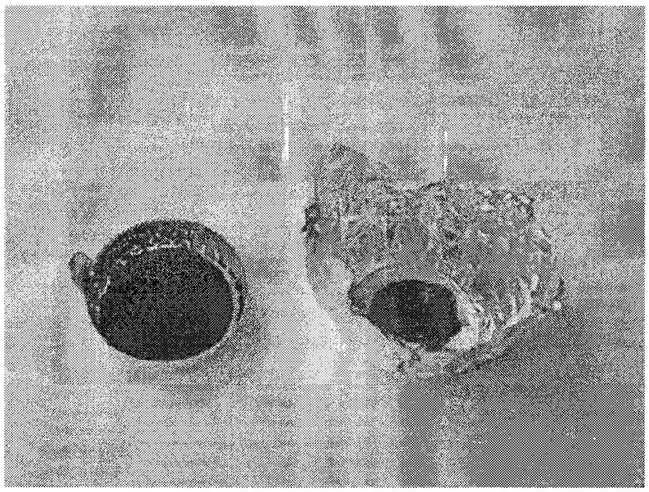

FIG. 3*a*—*Standard aluminum dish versus aluminum foil.*

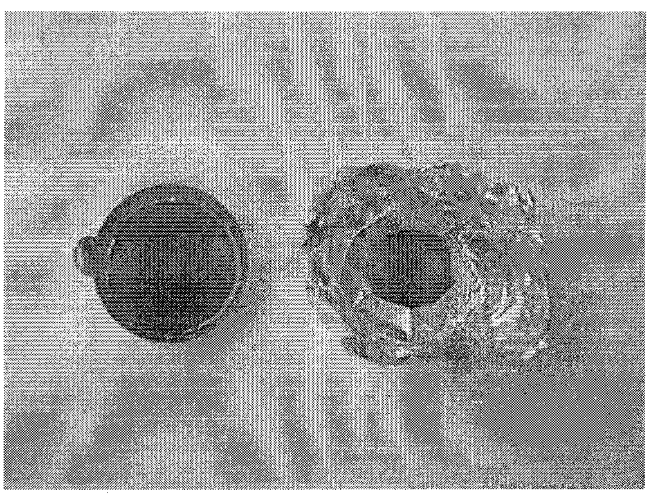

FIG. 3*b*—*Standard aluminum dish versus aluminum foil, top view.*

Determination of Curing Time for Asphalt Emulsions

In order to determine appropriate curing conditions, five aluminum dishes, each with 3 g of emulsified asphalt (CRS-2) fully covering its bottom area were heated for 24 h at 60°C. After 24 h, the weight of the dishes was determined and, subsequently, the dishes were reheated for another 24 h. The weight of each dish at the end of 48 h was determined and compared with the weight after 24 h curing. It was observed that the differences in weight between 24 and 48 h curing time was less than 0.01 g or approximately 0.3% by weight of emulsion. Table 1 shows the test results for curing time determination. Based on the results, a 24 h curing time at 60°C was selected for the remainder of the study. The amount of residual asphalt was found to be approximately 66% by weight of the asphalt emulsion (i.e., the ratio of asphalt to water in the CRS-2 emulsion is 2:1), which was to be expected. Although an asphalt emulsion was used in this study, it is possible to use an asphalt binder directly.

Determination of the Quantity of Solvent Used

Once the curing time was selected, the goal was to determine the quantity of solvent to be used. The same specimens from the previous step were used. It may be noted that after 48 h of heating, the residual asphalt in each dish was about 2 g. To each one of these samples, 1 g of solvent was added. Five solvents were used (S1–S4,

TABLE 1—*Test results for the curing time.*

Dish	Dish Weight (g)	Weight of Emulsified Asphalt (g)	Weight of Dish + Asphalt after 24 h Heating (g)	Weight of Dish + Asphalt after 48 h Heating (g)	Difference of Asphalt Weight Between 24 h and 48 h Heating (g)	Percent of Asphalt in Emulsified Asphalt (after 24 h Heating) (%)
1	0.994	3.049	3.031	3.023	0.008	66.55
2	0.996	3.041	3.033	3.025	0.008	66.72
3	1.000	3.037	3.031	3.021	0.010	66.55
4	0.997	3.035	3.029	3.018	0.011	66.59
5	0.994	3.045	3.039	3.028	0.011	66.80

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TABLE 2—*Test results for asphalt removed using 1 g of solvent.*

Dish	Type of Solvent	Weight of Solvent (g)	Weight of Dish + Asphalt after Washing and Heating (g)	Asphalt Removed (g)
1	S1	1.013	2.725	0.306
2	S2	1.007	2.755	0.278
3	S3	1.018	2.664	0.367
4	S4	1.006	2.826	0.203
5	Diesel	1.030	2.866	0.173

and diesel fuel)—with each dish having a different solvent. The asphalt was allowed to soak in the solvent for 5 min, then drained for 5 min (by keeping it upside down on an absorbent paper), and washed thoroughly under running water for five min. After washing, the samples were oven dried at 60°C for 15 h and weighed to determine the amount of asphalt removed. Results are presented in Table 2.

Three things need to be noted here. First, 3 g of CRS-2 emulsion (2 g residual asphalt) generated a fairly thick layer at the bottom of the dish. Due to this, it was decided to use 1.5 g of CRS-2 emulsion, which would result in 1 g of residual asphalt, equal to the weight of aluminum dish used. It was found that 1.5 g of CRS-2 emulsion fully covered the bottom of the dish. Second, 1 g of solvent was found excessive. Since the emulsified asphalt (CRS-2) weight was recommended to be half of the original 3 g, the solvent weight to be used was reduced to 0.5 g. This amount of solvent was found adequate to completely soak the asphalt. Moreover, it was noted that there was not a significant difference for asphalt being removed under the condition of 3 g CRS-2 emulsion with 1 g solvent versus 1.5 g CRS-2 emulsion with 0.5 g solvent. Third, with 3 g of asphalt emulsion, there was deposition of dissolved asphalt on the inner walls of the aluminum dish that adversely affected the calculation of exact amount of asphalt removed. Therefore, to reduce variability, 1.5 g CRS-2 emulsion with 0.5 g solvent was selected for the further investigation.

Determination of Solvent Exposure Time

To facilitate rapid evaluation of the solvents, the solvent exposure times of 1 and 5 min were used. However, the results for 1 min soaking time were highly variable and are not reported in this study. It is probable that longer exposure times would give different results in terms of the amount of asphalt removed. Considering the practical implications, it is very unlikely for work crews to soak their tools and equipment in the solvent for more than 5–10 min, especially at the end of the working day. Therefore, as a matter of practicality, 5 min soaking time was deemed to be reasonable and was used for the development of the standard test method.

Proposed Standard Procedure

Based on the results of the exploratory study, the following standard test method was proposed and used to quantify the efficacy of the solvents in removing asphalt:

- label each aluminum weighing dish and determine its weight
- apply 1.5 ± 0.05 g of emulsified asphalt (CRS-2) into the aluminum dish ensuring that the asphalt emulsion fully covers the bottom surface area of the dish. Care should be taken not to taint the walls of the aluminum dish while applying the emulsion coating
- heat the aluminum dish, with the asphalt emulsion, in a convection oven at 60°C for 24 h
- remove the dish after 24 h and cool it to room temperature (25°C) for 2 h. Determine the weight of the dish and calculate the weight of residual asphalt
- apply 0.5 ± 0.05 g of solvent into the dish by dropper. Make sure that the residual asphalt remains completely covered by the solvent for 5 min
- let the dish drain for 5 min by putting it upside down over absorbent paper, avoiding direct contact of the absorbent paper with the asphalt residue
- rinse the dish thoroughly for 5 min under running water, avoiding removal of undissolved asphalt by possible water pressure
- heat the dish at 60°C for 15 h to remove the traces of water completely
- weigh the dish to determine asphalt removed

The standard procedure outlined above is represented in Figs. 4*a–d* and 5*a,b*. Each of the 11 solvents, including diesel fuel, was tested with six replicates for each solvent to determine the amount

FIG. 4*a*—*Determine the weight of the dish.*

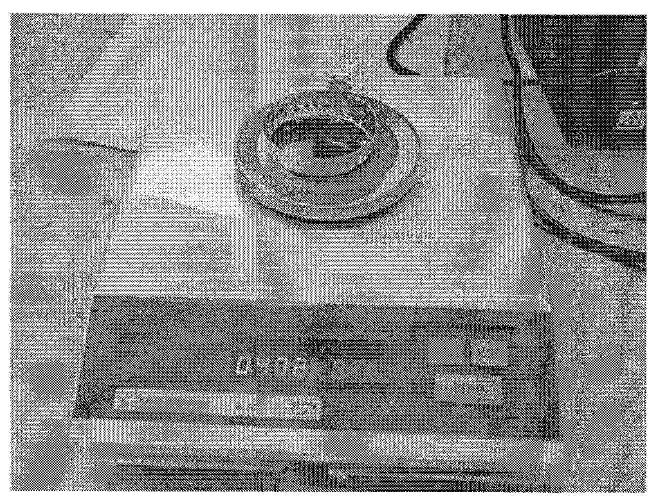

FIG. 4*b*—*Determine weight of asphalt emulsion.*

EXHIBIT B

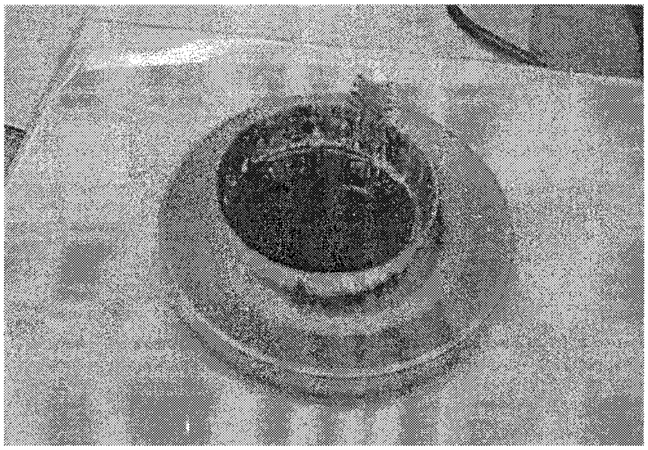

FIG. 4c—*Determine weight of residual asphalt after heating.*

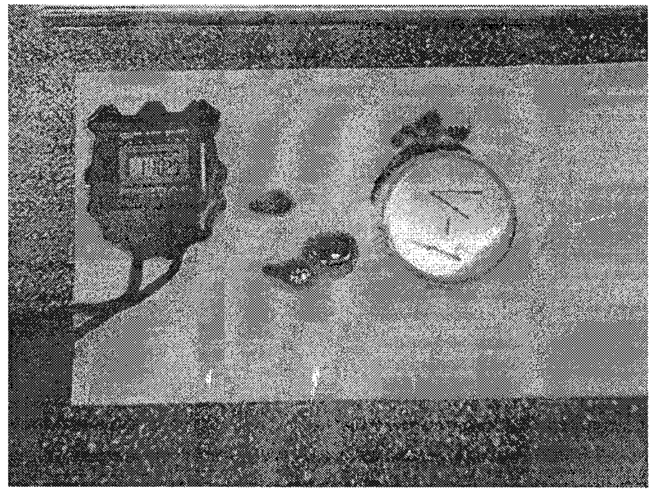

FIG. 5a—*Draining the dish for 5 min.*

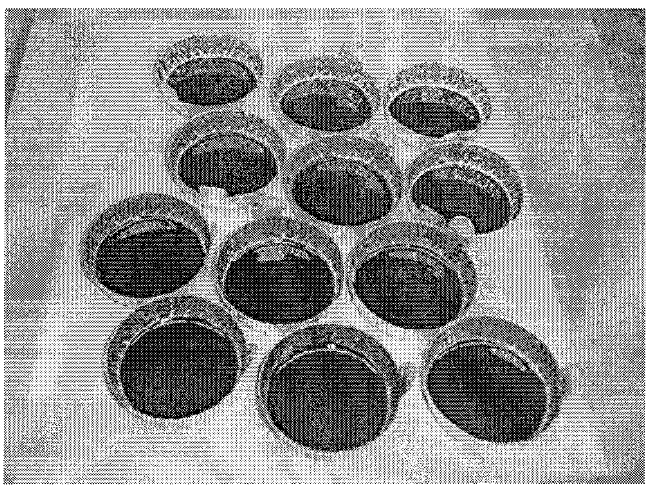

FIG. 4d—*Prepare replicates as required.*

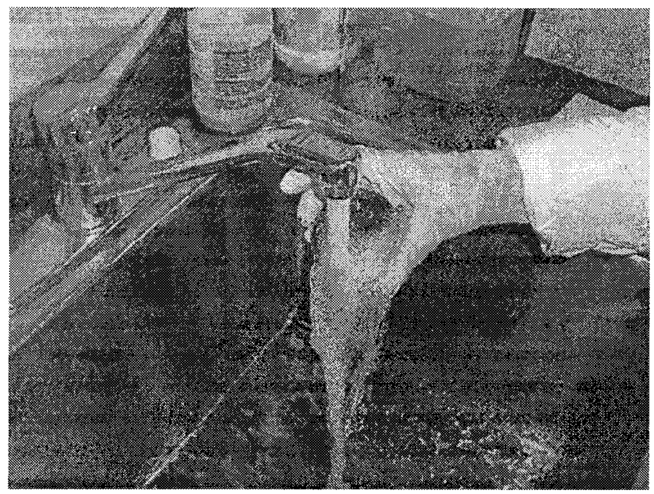

FIG. 5b—*Washing under running water.*

of asphalt removed. Test results are presented and discussed in the following sections.

Test Results

Table 3 summarizes the average data along with a statistical analysis of the results. Detailed data are presented in Ref. [5]. There are four parameters of interest for which average values are shown in Table 3: (1) absolute value of residual asphalt removed (column A), (2) percentage of asphalt removed (column C), (3) percent ratio of residual asphalt removed to the amount of solvent used (column D), and the (4) coefficient of variation for the amount of asphalt removed (column B). The third parameter is probably the most important as it directly shows the efficacy of the solvent used, i.e., the number of grams of asphalt removed in comparison to the number of grams of solvent used. Figures 6–8 are a visual representation of the same.

TABLE 3—*Data summary and results of statistical analysis.*

	Asphalt Removed (g)			
Solvent	Average (A)	CV% (B)	% (C = 100A ÷ Wt of Residual Asphalt)	Solvent Ratio (D = C ÷ Wt of Solvent)
S1	0.335	4.74	33	66
S2	0.291	5.13	29	57
S3	0.333	5.45	33	66
S4	0.224	6.48	22	44
Diesel	0.164	8.41	16	32
S5	0.004	55.75	0	1
S6	0.161	9.88	16	32
S7	0.354	6.04	35	70
S8	0.213	9.38	21	42
S9	0.165	6.39	16	33
S10	0.457	3.58	46	91

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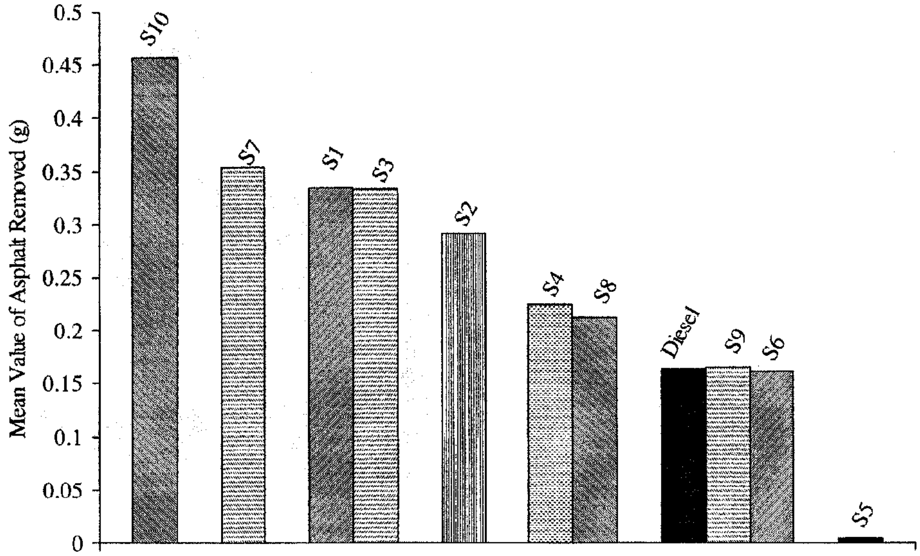

FIG. 6—*Comparisons of mean value of asphalt removed for 11 solvents.*

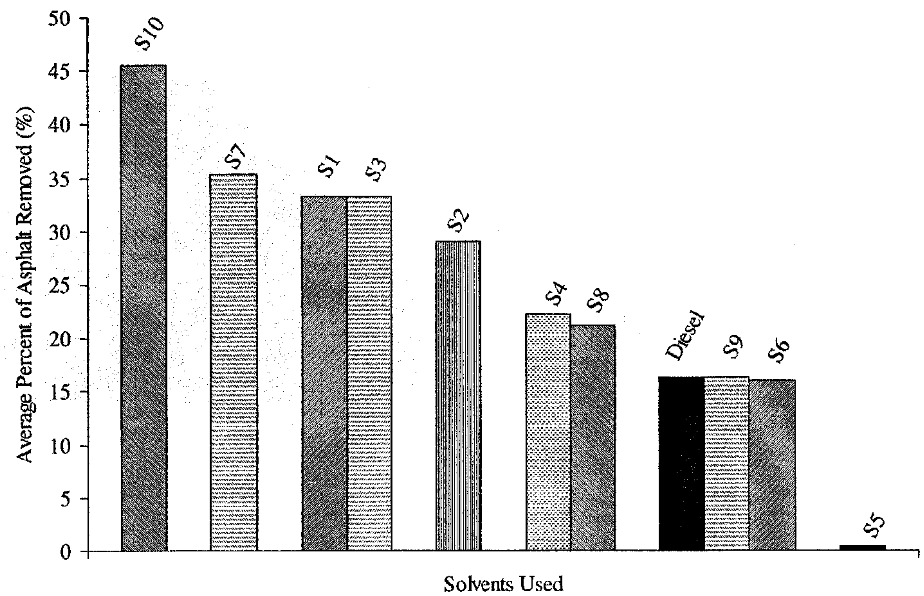

FIG. 7—*Comparisons of average percent of asphalt removed for 11 solvents.*

Table 4 shows the approximate cost per gallon of solvent (column E), and the coefficient of variation for asphalt removed per unit weight of solvent (column G). It can be seen from Table 4 that the CV values for asphalt removed vary from approximately 4–10% except for the S5 solvent. The 56% CV value for S5 solvent can be attributed to the fact that it was the least efficient of all the solvents in removing asphalt, and during testing the absolute amount of asphalt removed could not be meaningfully measured. In general, the CV values for the amount of asphalt removed are relatively small and acceptable for experimental data with so many variables (contact area, time of exposure, draining, washing, and heating) indicating that the standard procedure has low dispersion and high degree of repeatability. Efficiency with respect to diesel fuel (column H) represents the number of times (as a percentage) a given solvent is more efficient than diesel fuel. It is calculated as a percentage ratio of asphalt removed per unit weight of diesel fuel. Column I represents the cost efficiency with respect to diesel fuel. It is the cost of a given amount of solvent that can dissolve the same amount of asphalt as one gallon of diesel fuel. Figure 9 shows the cost efficiency with respect to diesel fuel. This parameter may ultimately be of use to assess the cost-efficiency of the solvents. Based on the asphalt removed as well as the cost-efficiency analysis, solvent S10 is the

EXHIBIT B

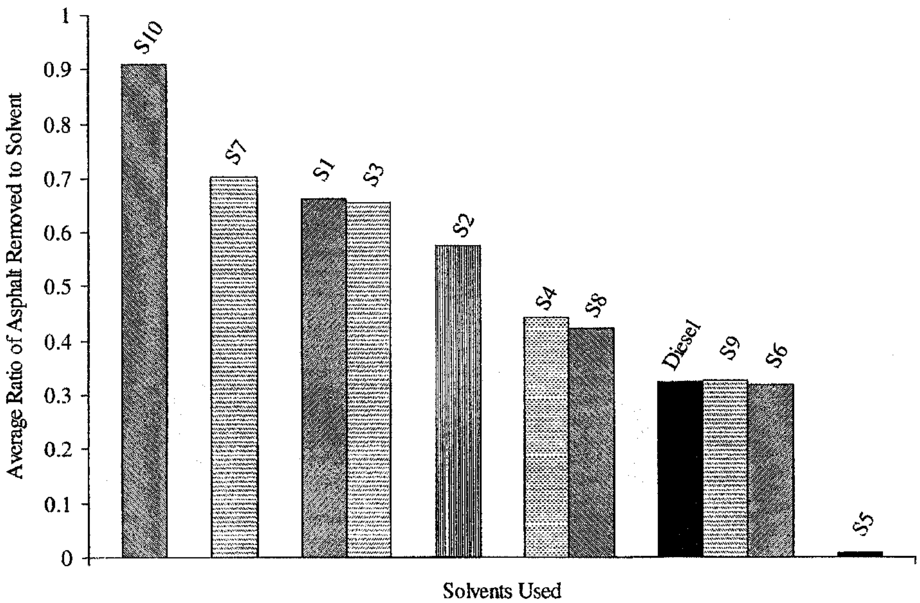

FIG. 8—*Comparisons of ratio of weight of asphalt removed to solvent for 11 solvents.*

TABLE 4—*Cost analysis.*

Solvent	Approx Cost per Gallon ($)[4] (E)	Asphalt Removed per Unit wt of Solvent (F = D ÷ 100)	Coefficient of Variation (G = CV(F))	Efficiency wrt Diesel (%) (H = 100F ÷ y[5])	Cost Efficiency wrt Diesel ($) ($I = 100^{E} \div F$)
S1	15.80[6]	0.662	5.26	206	7.67
S2	17.45[7]	0.574	5.24	178	9.80
S3	11.55[7]	0.655	5.92	206	5.61
S4	14.95[7]	0.443	6.19	138	10.83
Diesel	1.00	0.324	8.33	100	1.00
S5	...	0.008	56.49	3	...
S6	13.20[6]	0.319	10.41	100	13.20
S7	14.18[6]	0.703	6.01	219	6.48
S8	9.25[7]	0.422	9.69	131	7.06
S9	9.00	0.326	6.89	103	8.75
S10	13.85[6]	0.909	3.43	285	4.85

[4] Cost data obtained from NCDOT, except for S9 and diesel fuel where the data were obtained directly from the manufacturer.
[5] y = Asphalt removed per unit weight of diesel (Column F).
[6] Freight additional.
[7] Price includes freight.

most efficient. Although solvent S6 is as effective as diesel fuel, it is the least cost efficient solvent.

A statistical paired t-test was also performed on the data to evaluate if the differences in the asphalt removal capacity of the solvents are statistically significant. Assume that the solvency capacity of each solvent has a normal distribution $N(\mu, \sigma^2)$, where μ is the mean value for the solvency capacity, and σ^2 is the variance. In this experiment, only a sample about solvency capacity was available from the population without knowing the μ and σ^2. Therefore, comparison was made based on experimental samples' values with:

$\bar{x}$ = average value of lost asphalt, and

s = sample standard deviation.

Taking any two of the 10 solvents (Solvent S5 was not included in the analysis), assume the variance $\sigma_1^2 = \sigma_2^2$. Under significance level $\alpha = 0.1$, an F-test was performed to determine whether the hypothesis can or cannot be rejected. Based on the sample variance s^2, it was found that:

$$0.2 < F = \frac{s_1^2}{s_2^2} < 3.64 \tag{1}$$

If $F \geq F_{0.05}(5.5) = 5.05$ or $F \leq F_{1-0.05}(5,5) = 0.1980$, then the hypothesis should be rejected. For this case, the hypothesis could not be rejected, which indicated that the variance of any two samples could be taken to be equal.

Next, the t-test was performed to compare the mean value of a solvent with respect to diesel fuel under significance level of 0.05.

EXHIBIT B

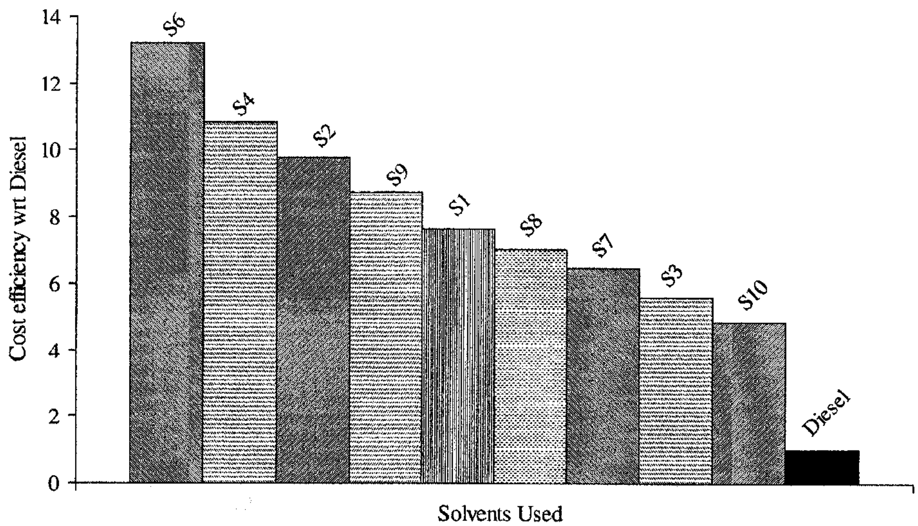

FIG. 9—*Cost efficiency with respect to diesel fuel.*

TABLE 5—*Results of statistical analysis t-test.*

Solvent	S1	S2	S3	S4	Diesel Fuel	S6	S7	S8	S9	S10
Diesel	< [−0.1904, −0.1522]	< [−0.1456, −0.1087]	< [−0.1899, −0.1485]	< [−0.0785, −0.0422]	. . .	= [−0.017, 0.0213]	< [−0.2131, −0.1669]	< [−0.0712, −0.0271]	= [−0.0171, 0.0144]	< [−0.3126, −0.2737]

Results are listed in Table 5. The signs ">," "<," and "=" mean that the solvency capacity of a given solvent is "stronger," "weaker," or "equal," respectively, in comparison to diesel fuel. The "C. I. = [a, b]" means that the solvency difference of a given solvent and diesel fuel will fall into the interval [a, b] with 95% confidence. For example, in comparison to diesel, solvents S6 and S9 are both equally good and their performance is statistically not different from diesel fuel. All other solvents show statistically superior performance compared to diesel fuel in terms of the amount of asphalt removed.

Conclusions and Recommendations

Figures 6–8 show the rating of the solvents based on the absolute average, percent asphalt removed, and average ratio of asphalt removed to solvent. Figure 9 shows the cost efficiency of each solvent with respect to diesel fuel. Irrespective of the parameter used, solvent S10 performed the best and was found to be the most cost-efficient solvent. Solvent S6, which was equal in performance to diesel fuel, was found least cost-effective. Solvent S5 did the poorest and, for all practical purposes, did not clean the asphalt. The remaining solvents were found statistically better or equally good as compared to the diesel fuel.

The standard test procedure developed in this study gave results that are quantifiable, reliable, and have good repeatability with low coefficient of variation. This proposed test procedure provides a method to evaluate the efficacy of asphalt removing solvents. However, it should be noted that a single operator did all testing. Further investigation is necessary to study the effect of multiple operators and multiple laboratories. The asphalt used in this study was a CRS-2 emulsion in an "unbroken" state. However, it is also possible to first break the CRS-2 emulsion separately, and then weigh 1 g of residual asphalt directly into the aluminum dish. This would eliminate the heating of the dish for 24 h. However, this procedure was not studied in this investigation.

Acknowledgments

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[2] "Fisher*brand*™ Aluminum Weighing Dishes with Fluted Sides," Fisher Scientific (URL: http://www.fishersci.com) Fisher Catalog number 08-732.
[3] "Identification and Listing of Hazardous Waste," *Code of Federal Regulations, Title 40, Part 261*, (URL: http://www.access.gpo.gov/nara/cfr/waisidx_02/40cfr261_02.html) Revised July 2002.
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EXHIBIT B

Appendix

The solvents were received from the North Carolina Department of Transportation. Compositions, being proprietary, are not known to us. A table describing the trade names of the solvents is as follows:

Label	Trade Name	Manufacturer	Address
S1	Orange	CIM Supply, Inc	4813 Sardis Rd, Ste 4, New Kensington, PA 15068 (724) 335-1798
S2	Pave-Pro	Chemtek, Inc	PO Box 86, Yanceyville, NC 27379 (336) 694-1561
S3	Citrus-60	Major Chemical Company	PO Box 21226, Charlotte, NC 28277 (704) 552-4722
S4	Pave-RX	Chemtek, Inc	PO Box 86, Yanceyville, NC 27379 (336) 694-1561
S5	Eliminator TA	Continental Research Corp	PO Box 15204, St. Louis, MO 63110 (800) 325-4869
S6	Orange Slicker	Orange Products Corp	3020-B Thurston Avenue, Greensboro, NC 27406 (336) 698-0000
S7	Big-Orange E	ZEP Manufacturing Company	Atlanta, GA 30301
S8	HTF-959B	Terpene Technologies Southeast, Inc	Ray Chestnut, (336) 855-7410
S9	Bean-E-Do	Franmar Chemical, Inc	PO Box 97, Normal IL 61761 (309) 452-7526
S10	Citrus-Sol-PlusE	Uni-Chem Corporation of Florida	1200 Clint Moore Rd, Suite 4, Boca Raton, FL 33487 (561) 995-2121

EXHIBIT A

Robert Ernest Troxler, PhD

EDUCATION:

- **Doctor of Philosophy in Electrical Engineering**, Georgia Institute of Technology. DISSERTATION TITLE: *Magnetic Control of Superconducting Phase-Shifters with Experimental Verification.* December, 1992.
- **Master of Science in Electrical Engineering,** Georgia Institute of Technology June, 1986.
- **Bachelor of Science in Electrical Engineering,** North Carolina State University, December 1983.

EXPERIENCE:

- **Director of Advanced Technologies** (March 1998 – present), Troxler Electronic Laboratories, Inc., RTP, NC.
- **Research Engineer** (January 1996 – March 1998), Troxler Electronic Laboratories, Inc., RTP, NC.
- **Director North Carolina State University Engineering Foundation**, (April 1998 – present)
- **Consultant to Electromagnetic Theory** (February 1993 – January 1996)
- **Design Engineer** *(December 1983 - September 1984)* Troxler Electronic Laboratories, Inc., Research Triangle Park, NC. Designed microprocessor based portable equipment for nondestructive evaluation of materials incorporating nuclear isotopes.
- **Awarded fellowship** sponsored by Electromagnetic Sciences Incorporated – Atlanta, Georgia. (June 1992 – December 1992)
- **Graduate Work** *(1984-1992)*

  June 1989 –June 1992: NASA Fellow (Graduate Student Researchers Program), *Marshall Space Flight Center, Huntsville,* Alabama. *Georgia Institute of Technology, Graduate Research (Teaching) Assistant,* Georgia Institute of Technology, Atlanta, Georgia. The objective of this research was to investigate the feasibility of controlling the phase velocity of thin film superconducting delay lines with a biasing magnetic field or transport current.

  November 1990-November 1991: On site at Marshall Space Flight Center (Space Science Laboratory) to perform the experimental part of thesis. Participated in a flight experiment to study the effects of atomic oxygen on space materials. Deposited many thin films for this experiment.

  September 1984 - June 1989 Graduate Research (teaching) Assistant, Georgia Institute of Technology, Atlanta, GA. Taught Graduate Mircrowave Laboratory utilizing an HP-8510 Automatic Network Analyzer. Researched procedures of cryogenic microwave measurements. Researched possible microwave applications of High Temperature Superconductors supported by internal “seed funds”. Wrote a laboratory procedures manual consisting of passive microwave devices for the Graduate Microwave Laboratory. Researched the possibility of using ferrite loaded images lines as phase-shifters.

EXHIBIT A

PATENTS ISSUED:

7,389,199 Method of Determining a Dimension of a Sample of a Construction Material

7,376,530 Paving Related Measuring Device Incorporating a Computer Device

7,239150 Pavement Material Microwave Measurement Methods and Apparatuses

7,034,695 Large Area Position/Proximity Correction Device with Alarms using GPS

6,990,848 System and Method for Determining Material Properties of Samples

6,915,216 Measurement Device Incorporating a Locating Device and Portable Computer

RE38,910 Low Activity Nuclear Density Gauge

6,579,500 Apparatus and method for determining weight loss of a heated material

6,567,498 Low activity nuclear density gauge

6,492,641 Apparatus and method for gamma-ray determination of bulk density of samples

6,442,232 Thin layer nuclear density gauge

6,440,746 Method and apparatus for analyzing asphalt content

6,436,718 Apparatus and method for determining weight loss of a heated material

6,369,381 Apparatus and method for calibration of nuclear gauges

6,310,936 Thin layer nuclear density guage

6,054,323 Method and apparatus for analyzing asphalt content

OTHER PUBLICATIONS:

- G.P. Rodrigue, R.E. Troxler, and J. Smart, *Microwave Measurements*, Laboratory manual for Georgia Institute of Technology.
- R.E. Troxler and G.P. Rodrigue, *Analysis of Ferrite Loaded Rectangular Image Guide*, Prepared for The Army Ballistic Missile Defense Advanced Technology Center, Huntsville Alabama 35807, October 10, 1985.
- R.E. Troxler, High Temperature *Superconducting Phase-Shifters,* A Successful Proposal to NASA's Graduate Student Researchers Program, Marshall Space Flight Center, Janurary, 1989.
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